Research Report R80-1

A MICRODEBONDING TEST FOR IN-SITU FIBER-MATRIX BOND STRENGTH AND MOISTURE EFFECTS

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February 1980

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
Cambridge, Massachusetts 02139

-- 3 OF 3
-- **NTIS DOES NOT HAVE THIS ITEM**
-- 1 - AD NUMBER: D418404
-- 2 - UNCLASSIFIED TITLE: A MICRODEBONDING TEST FOR IN-SITU FIBER-
-- MATRIX BOND STRENGTH AND MOISTURE EFFECTS,
-- 10 - PERSONAL AUTHORS: MANDELL,J. F. ;CHEN,S. H. ;MCGARRY,F. J. ;
-- 11 - REPORT DATE: FEB 11, 1980
-- 12 - PAGINATION: 6P
-- 20 - REPORT CLASSIFICATION: UNCLASSIFIED
-- 21 - SUPPLEMENTARY NOTE: IN PROCEEDINGS: 35TH ANNUAL CONFERENCE,
-- RISING TO THE CHALLENGE OF THE '80S', 4-8 FEB 1980, NEW ORLEANS, LA.
-- SPONSORED BY SOCIETY OF THE PLASTIC INDUSTRY, REINFORCED PLASTICS/
-- COMPOSITES INSTITUTE. SECTION NO. 26-D. (SEE PL-34705).

-- 22 - LIMITATIONS (ALPHA): APPROVED FOR PUBLIC RELEASE; DISTRIBUTION
-- UNLIMITED. AVAILABILITY: SOCIETY OF THE PLASTICS INDUSTRY, INC.,
-- 355 LEXINGTON AVE., NEW YORK, N.Y. 10017.
-- 33 - LIMITATION CODES: 1 24
-- ******
-- 4 OF 4

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A Microdebonding Test for In-Situ Fiber-Matrix Bond Strength and Moisture Effects

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Abstract

The paper describes a test technique which gives a quantitative measure of the in-situ fiber/matrix bond strength in composites. The test involves the compressive loading of a fiber or region of fibers on a polished specimen surface to produce debonding. Results are given for the debonding load for glass, aramid and graphite/epoxy composites. The change in debonding load is also followed as the interface degrades during moisture conditioning at different points through the thickness. Refinements are needed to simplify interpretation of the debonding force in terms of interface shear strength, and to make the test more reproducible.

Introduction

The purpose of this continuing study is to develop a mechanical test for quantitative measurement of the fiber/matrix interface strength in fiber composites. It is of particular interest that the test be sensitive to the effects of moisture on the interface strength.

Tests to elucidate the quality of the interface have been used throughout the history of fiber reinforced plastics. These may be divided into tests on model systems which do not use the composite in its usual condition, and tests on the actual composites of interest. The model systems are limited because they cannot give complete information about the condition of an actual composite. The commonly used adhesives tests (1) may be viewed as model systems if the appropriate adherend, adhesive, and surface treatment are used. Models more similar to composites have used single fibers embedded in resin (2,3) to obtain the interface shear or tensile strength. These tests may also be used with strands or yarns, but they depend on the matrix remaining elastic until debonding occurs, which is not satisfied in all of the materials studied here, particularly under wet conditions. Another test which can be used with ductile resins involves the breakdown of the fiber into shorter lengths as a function of matrix strain (4).

Macroscopic strength tests provide data on actual composites, but require interpretation to deduce even the qualitative state of the interface. The failure of composites in macroscopic tests is a complex process, and is not necessarily

caused by failure of the interface. However, macroscopic tests for matrix/interface dominated properties such as parallel shear (5) and transverse tension (6) have found utility in providing quantitative data for relative changes in the interface strength. In this study, it was observed that these properties often reflect the pre-existence or development during loading of discrete cracking regions. Although the resistance to such discrete cracks may reflect the interface strength, their presence tends to dominate the mechanical properties after their formation, preventing any sensitivity of the test to the great majority of interfaces which are still intact. Indeed, efforts in this study to find a macroscopic mechanical test in which most of the fibers could be made to debond in glass/epoxy materials were unsuccessful. Fracture surfaces from macroscopic tests may give information about the interface strength as related to the debonded fiber length (7). This information is also difficult to interpret, since it may depend on the local fiber strength and flaw distribution, matrix ductility, mode of crack propagation, etc.

The foregoing demonstrates a need for an interface test which: 1) can be run on a typical fiber composite, 2) clearly measures the failure point of the interface, and 3) can be applied to selective regions remote from cracks and voids. It is desirable to determine the actual strength of the interface, rather than some parameter such as the average stress in the composite to cause interfaces to fail. The microdebonding

test described here appears to satisfy most of these requirements if it can be made to give reproducible results, and if the stresses can be analyzed with sufficient accuracy. This paper provides an indication of the feasibility and potential of such a test method, but further development and experience are required before it can be proposed as a practical and reliable way to determine the interface strength.

EXPERIMENTAL METHODS

The microdebonding test is illustrated in the schematics of Figure 1. The following are the characteristics of the test as it has been applied in this study:

1. A metallographically polished surface of the material is prepared, with the fibers normal to the surface.
2. A steel probe is brought into contact with the surface under a known load.
3. The probe is removed, and the surface is inspected microscopically for fiber/matrix debonding.
4. Successively higher loads are applied on the same area until debonding of one or more fibers is observed, or a new area may be used for each test.

As indicated in Figure 1 in two dimensional profiles, several probe-tip geometries were studied, each giving a different debonding pattern. The axial force to cause debonding is defined in different ways, depending upon the probe geometry. For example, some tests use the force for the earliest de-

tectable debonding, usually a single fiber or two fibers; tests with other probes use the development of debonding around a ring of 6-7 fibers as the critical force. The choice of probe geometry and debonding pattern are major parameters in this exploratory study. Probes were produced by machining and/or polishing either conventional sewing needles or drill rod stock.

Tests were conducted in a Vickers microhardness tester which was altered to use the steel probes. The loading mechanism also was altered to allow increments of one gram loads with the initial load from the apparatus offset by a counterbalance. Unless otherwise noted, the load was applied to the specimen for 45 seconds at each step. The time of load application has an effect on the results, but was not systematically studied.

The material used for most of the study was Scotchply Type 1009 unidirectional E-glass/epoxy (3M Co.), 1.3 mm. thick. Other tests were run on T300/5208 graphite/epoxy and Kevlar 49 aramid/epoxy with an unidentified matrix. Specimens were moisture conditioned by soaking in distilled water at various temperatures, followed by oven drying at 95°C for 24 hours (including unconditioned controls). Then the specimens were potted in a polyester compound and wet sanded and polished with alumina following standard metallographic procedures. Microdebonding measurements were made as near to the center (midthickness) of the specimen as possible except as noted. All testing was done in an air-conditioned laboratory with uncontrolled humidity. The number of test replications and the loading increments will be discussed later.

RESULTS AND DISCUSSION

General Test Characteristics

The schematics in Figure 1 and the micrographs in Figure 2 indicate the most definitive observation of the study: it is possible to produce controlled fiber/matrix debonding by loading fibers or small groups of fibers on the polished surface of a composite specimen. Any fiber or region can be selected by viewing in a microscope and then loaded in increments until it is observed to debond, thus providing a quantitative measure of its resistance to debonding. Interface failure is the first permanent damage observed in most cases, so it can be assumed that debonding occurs under conditions approximating elastic behavior. This might not be true if more ductile resins or wet resins were used, but debonding was the first damage observed for all cases reported here.

Other encouraging results are given in Figures 3 and 4. Figure 3 indicates that the test can be applied to a variety of important composites (interpretation of the relative debonding loads will be discussed later). Both Figures indicate that a debonding force can be determined with acceptable scatter, and that results are reproducible if the same probe is used. The probes used were of the type illustrated in Figure 1c. Each point represents loading to the given force at ten positions on the specimen surface. No damage to the probes could be observed in a scanning electron microscope after many load applications in this range. Load increments of one gram were used for most cases.

Comparison of the glass/epoxy data in Figure 3 with Figure 4 also illustrates the most difficult problem with the test: changing the probe or the alignment of the apparatus usually shifts the range of debonding forces, although the relative performance of different materials is preserved. Thus, results for a number of cases can be compared only if the same probe and apparatus positioning are used, or if comparisons are indexed to a standard material. Overloading of the apparatus can damage the probe or change the alignment, so one probe condition is difficult to preserve indefinitely.

General experience with the probe shapes given in Figure 1 (a-d) is as follows (fibers are approximatley 8μm in diameter and serve as a scale):

(1a) The rounded tip similar to an as-received conventional sewing needle can be used to produce single fiber debonding, or debonding over a larger domain if higher loads are used. When single fiber debonding is produced, the load appears to be distributed over a larger domain than just the debonded fiber, so analysis is difficult.

(1b) The flat probe is produced by lapping a pointed drill rod on a fine stone. It produces debonding around a ring at its periphery but requires such precise alignment to avoid uneven loading that it has not been successful in practice.

(1c) The slightly rounded shape produced by the sanding and polishing of a needle embedded in resin gives debonding over a region of several fibers, depending on the force. As this shape approaches (1b), the debonding occurs around the periphery only, but alignment becomes more difficult.

(1d) The sharp tip produced by grinding a drill rod (Figure 1d) appears to give good results for single fiber debonding, and it can load only a small area at the center of the fiber. However, the debonding loads are less than one gram for degraded glass/epoxy, and the present apparatus cannot function accurately in this range. A new one is under development and it should provide the required accuracy. This type of loading should be most easily analyzed to give an interfacial shear strength if the debonding load is known. Alignment should also be less critical for this probe.

Moisture Degradation

The microdebonding resistance was determined for glass/epoxy specimens which had been conditioned in distilled water at several temperatures. The specimens were dried before testing, so changes in debonding resistance represent permanent degradation. The first batch of material, Batch A, was conditioned in 95°C water. The moisture gain is indicated in Figure 5, as is the moisture loss during subsequent drying (conditioning started with material which had not been initially

oven dried). The microdebonding load was determined for all conditions with the same probe and alignment. The probe used was similar to Figure 1(c), and the load was that required to consistently produce debonding over a region of 6-7 fibers near the mid-thickness. Approximately 20 tests were required to define each point. The debonding strength decreased with log exposure time in an approximately linear fashion. The transverse flexural strength measured in three-point bending following ASTM D-790 showed a trend given by the dashed line on Figure 5. The initial drop, followed by a flattening trend is similar to that reported elsewhere (5). The second drop in strength was associated with the development of discrete cracking regions during conditioning.

A second batch (B) of the same material was conditioned in 23, 60, and 95°C distilled water, dried, and tested with a probe similar to Figure 1(a). Figure 6 gives the moisture gain curves for these specimens, and Figure 7 gives the microdebonding load. Approximately 20 tests were run near the mid-thickness for each point, to determine the load to produce debonding on one or two fibers. The trend of the 95°C data is similar to that in Figure 5 for multi-fiber debonding with a different probe geometry. Much less loss in bond strength is observed for the other conditioning temperatures, at which specimens do not appear to approach equilibrium moisture content at the 300 hour maximum test time. The local moisture content near mid-thickness where the tests were run was not determined.

A major asset of the microdebonding test is the use of a small region of material so the interface condition can be determined at various positions through the thickness or very close to holes. Figure 8 gives the variation in microdebonding strength with distance through the thickness for three conditioning times of Batch A, again using approximately twenty tests to define each point. As expected, the gradient of bond strength with position is greatest at short conditioning times, with less difference in the control and at long times, as equilibrium is approached. After 100 hours conditioning, both the material at the center and the material near the surface have decreased to approximately 45% of the control value at that point. Thus, the degradation of the bond strength at any point may be more sensitive to the maximum moisture content reached than to the time at the maximum moisture content. However, more work is required to establish this observation beyond doubt.

Discussion

To date, the results obtained with the microdebonding test suggest that it can give a direct measure of the in-situ interface strength, and it may provide significant data not available from other tests. An unambiguous interface strength property would be of value to the composites industry for material development, quality control, research, application development, and failure analysis. However, a number of refinements and further studies are necessary before the test can be considered reliable and valid.

The most pressing requirement is for a probe and apparatus which are reliable and reproducible. This was discussed earlier, and work is in progress to develop a more sensitive apparatus using the probe geometry in Figure 1(d). There do not appear to be any fundamental limitations in terms of microscope, load measurement, or precision movement which cannot be satisfied by standard laboratory equipment.

If it can be performed reliably and reproducibly, the test will be of use even if the debonding load is all that can be determined. However, an analysis which could give the interface strength as a function of the debonding load, geometry, and material elastic constants would make the test even more useful. The analysis of contact problems of the type in Figure 1 is complex, involving changes in contact area, interaction between probe and specimen distortion, and singularities both at the edge of the contact area and possibly at the fiber/matrix interface on the surface. Fiber distribution and spacing could also be important if discrete fiber and matrix regions are considered in the model.

A number of unpublished finite element solutions using various assumptions about material homogeneity and loading have been obtained. While simple relationships are difficult to identify in the results, it is clear that the shear stress along the interface is the more important stress component since the stress normal to the interface is always compressive. Less complex results may be forthcoming from the single fiber loading scheme in Figure 1(d) if the loading occurs over a small area near the center of the fiber, and a

single fiber model is used, ignoring or smearing neighboring fibers. Here the nature of the local contact problem at the probe may not dominate the stresses at the interface where failure occurs. Micromechanics solutions for fiber pull-out or fiber ends (8,9) usually give an interface shear stress which is proportional to the fiber axial stress and to the ratio $(G_m/E_f)^{1/2}$, where G_m is the matrix shear modulus and E_f is the fiber Young's modulus. Such a data reduction factor may prove justified for the single fiber loading case, but should not be applied to other geometries or to the data in Figure 3.

Another complication in the test is the use of a polished surface, and the measurement of the bond strength very close to the surface. Qualitative experience to date has not indicated that sensitivity of the test to surface preparation or local moisture gradients due to surface drying, etc., are a major problem, but a systematic study of these effects has not been made. Residual stress effects near the free surface are also a concern. Micromechanics analysis of similar problems (10) suggests that the residual interface shear stress should be relatively low compared to the stress normal to the interface; the normal stress is of less concern here because it is compressive in the debonding test.

The present test procedure is time consuming because the load is applied incrementally, with microscopic observation at each step to detect the onset of debonding. Unfortunately, the debonding event does not change the stiffness of the system much and could not be determined from

a load-deflection curve. Other methods of detection such as electrical or ultrasonic will be attempted in the future. The test would be much more convenient if such a method to detect debonding could be established, so the load could then be continuously increased in a single test.

CONCLUSIONS

The microdebonding test provides a direct measure of the bond strength in a fiber composite. The test can be used to determine bond degradation during moisture exposure as well as the distribution of bond strength through the thickness. A number of refinements are necessary before the test can be used to give precisely defined, reproducible interfacial shear strength values, particularly for materials with differing elastic properties.

ACKNOWLEDGEMENT

This work was supported by the Army Materials and Mechanics Research Center through contract DAAG-46-77-C-0770.

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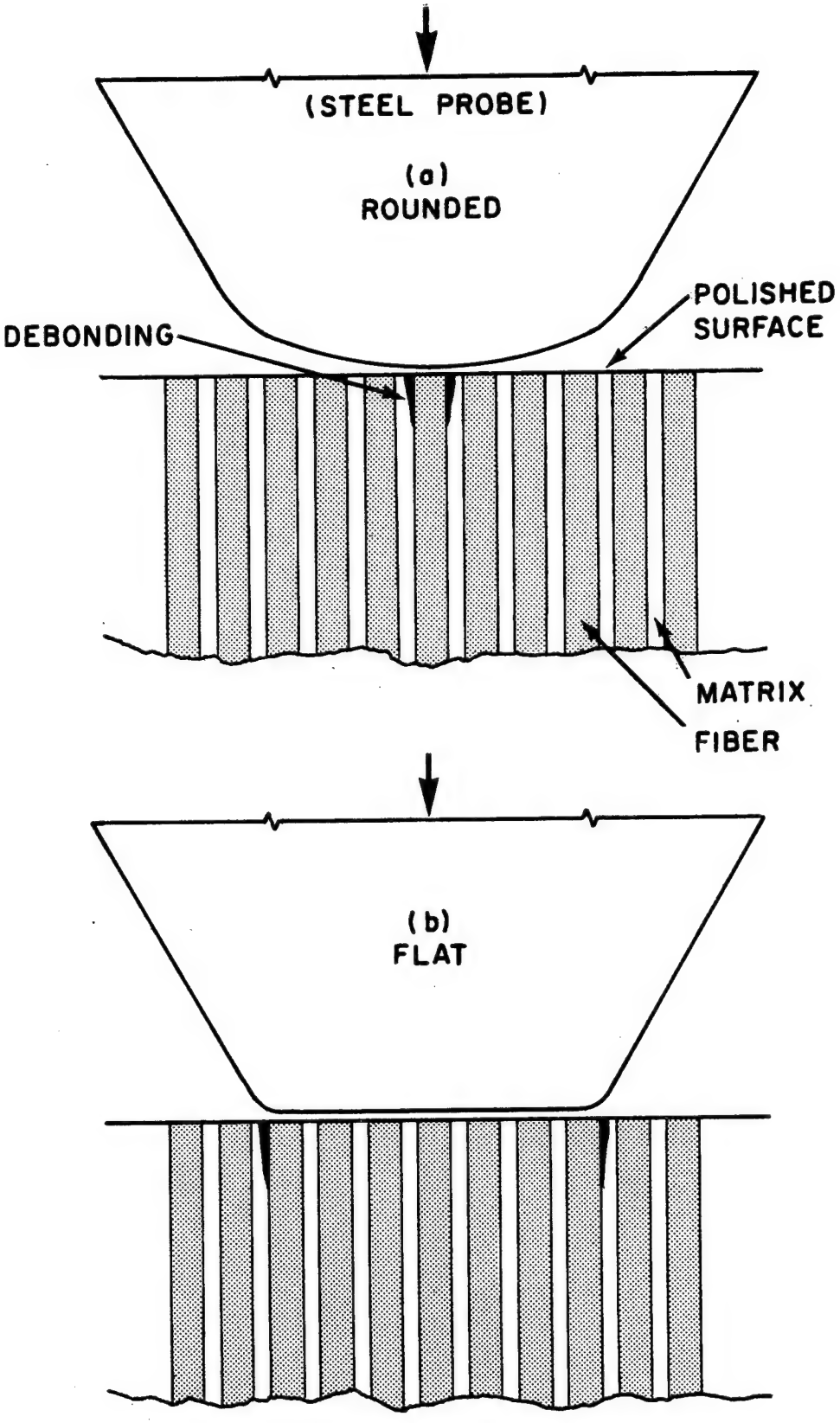
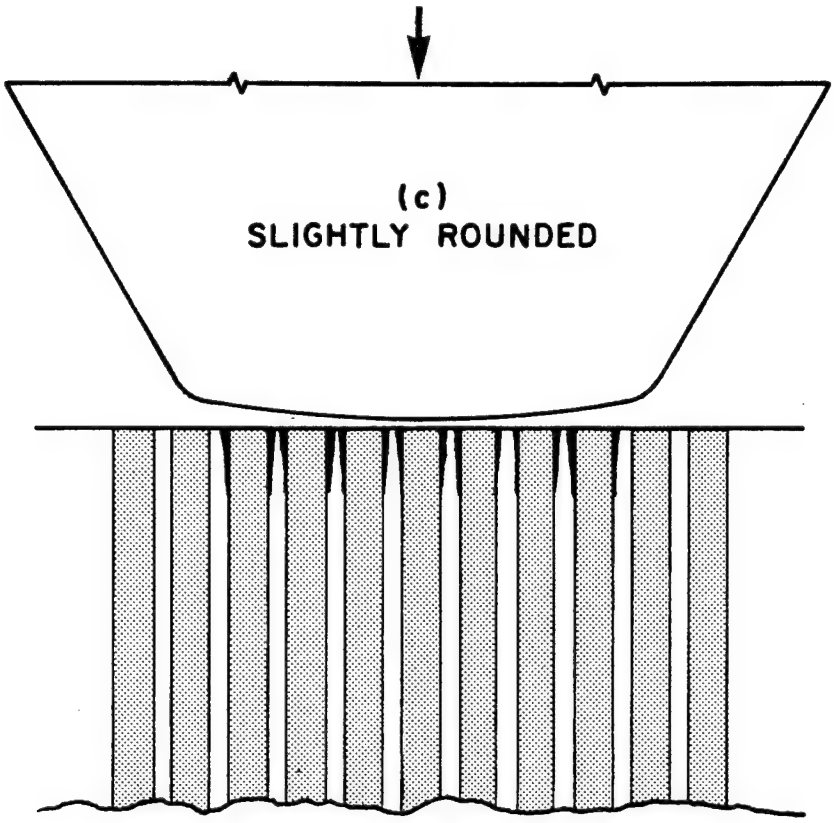


FIGURE 1.

TYPICAL PROBE GEOMETRIES AND ASSOCIATED DEBONDING PATTERNS.



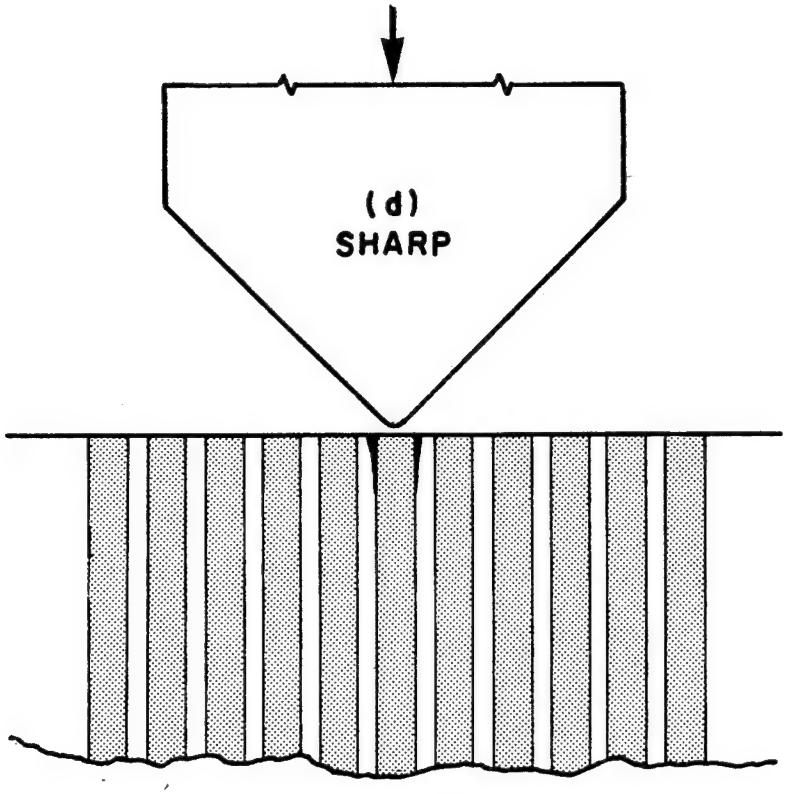


FIGURE 1 (continued).

TYPICAL PROBE GEOMETRIES AND ASSOCIATED DEBONDING PATTERNS.

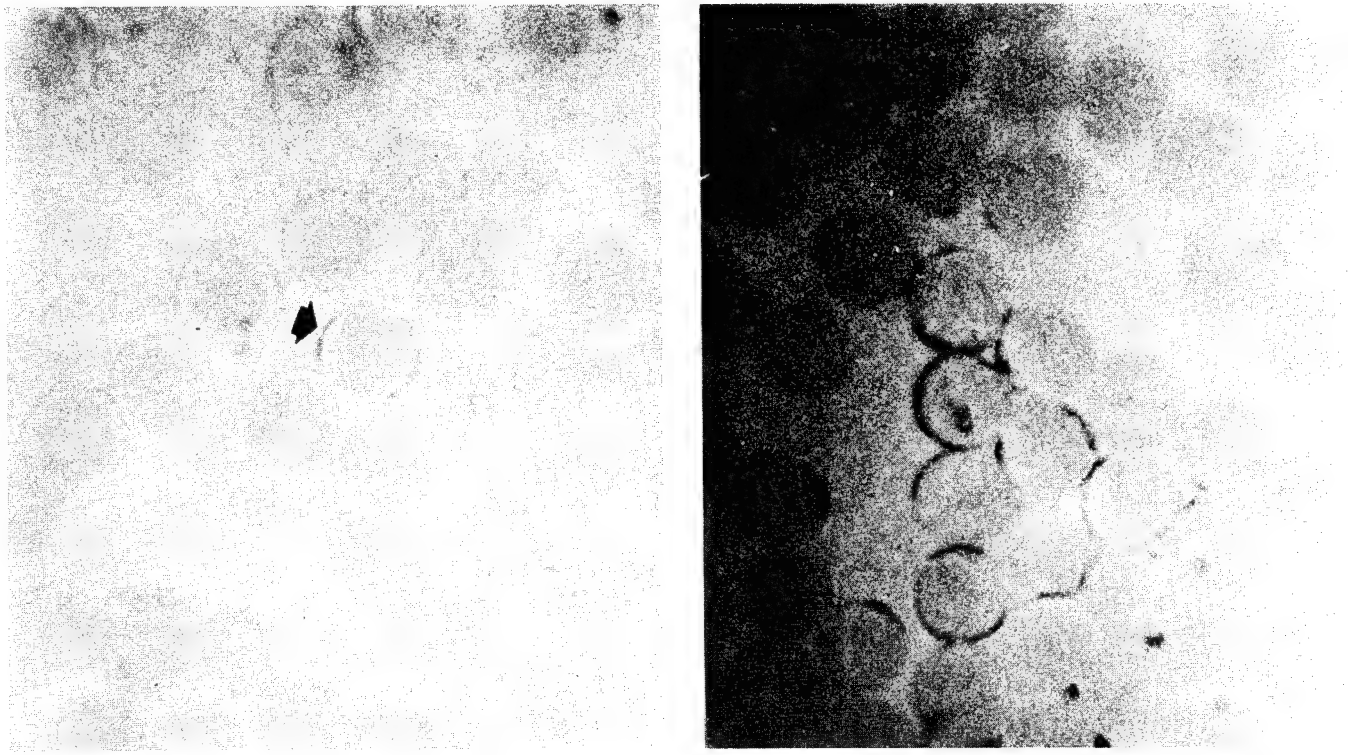


FIGURE 2. SINGLE FIBER (LEFT) AND MULTIFIBER DEBONDING ON GLASS/EPOXY SURFACE (FIBER DIAMETER = 8 μm)

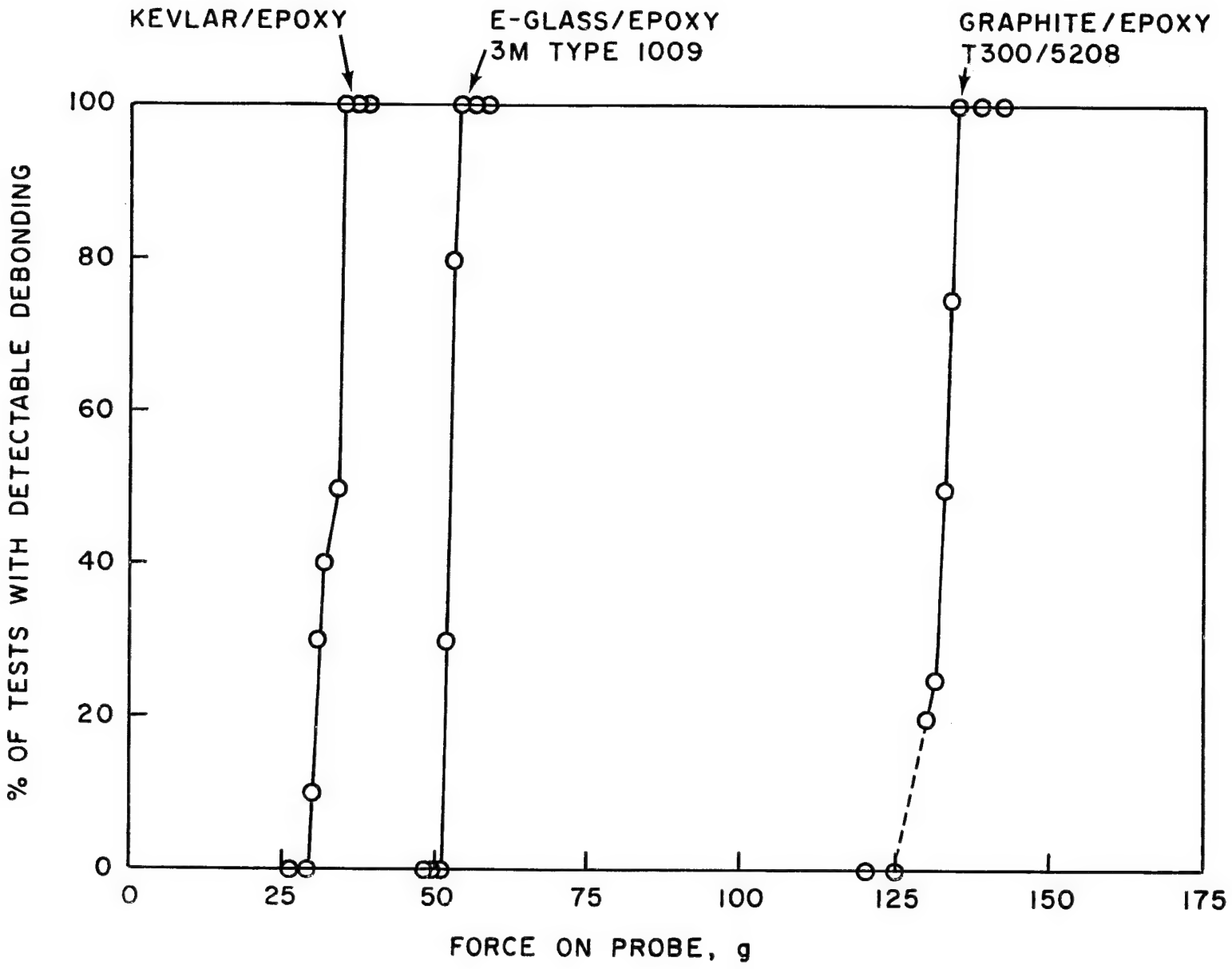


FIGURE 3.

MICRODEBONDING RESULTS FOR KEVLAR, E-GLASS, AND GRAPHITE FIBER/EPOXY USING SAME PROBE.

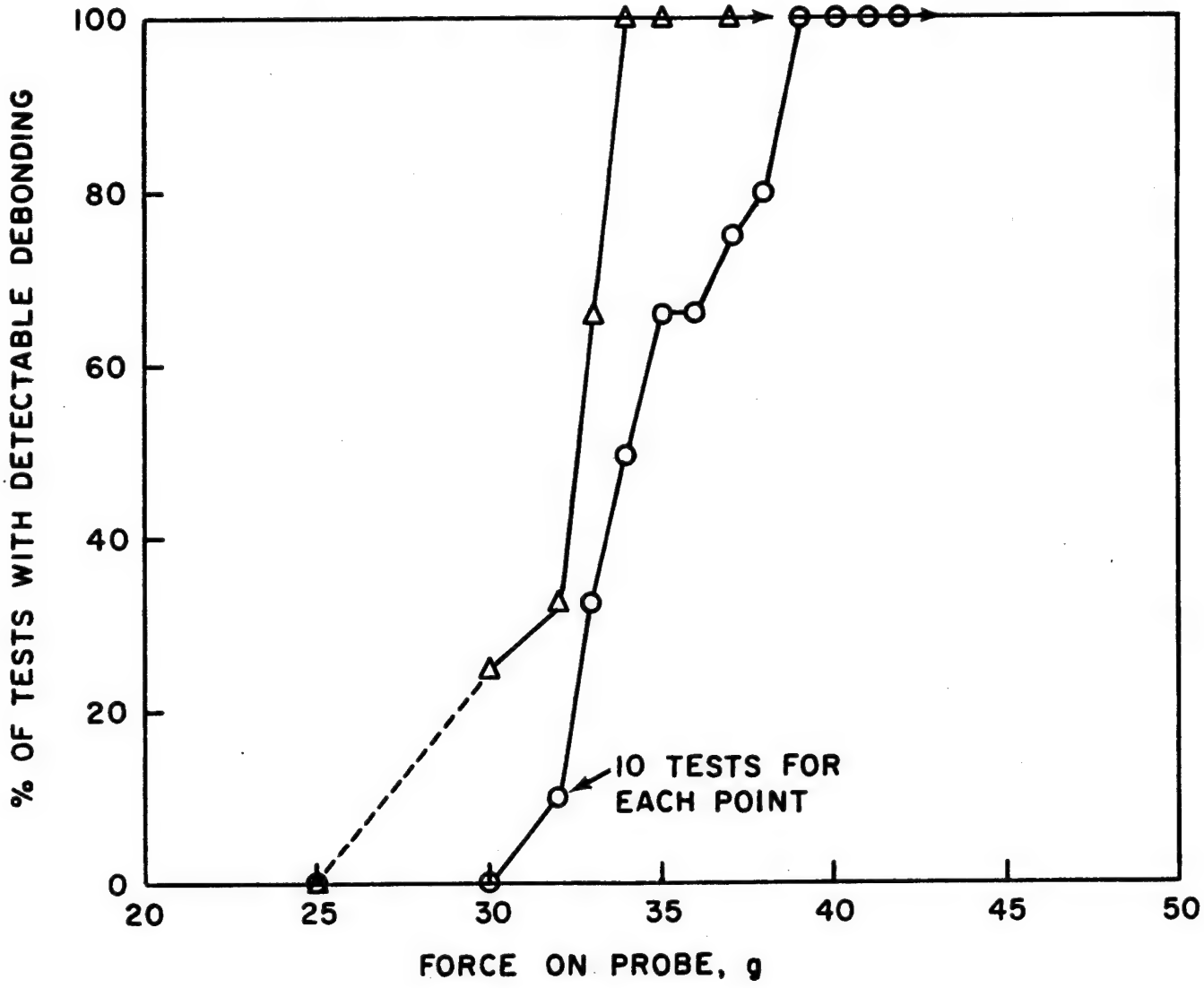


FIGURE 4.

MICRODEBONDING DATA FOR TWO SPECIMENS OF THE SAME PLATE OF TYPE 1009 E-GLASS/EPOXY; SAME PROBE FOR ALL TESTS.

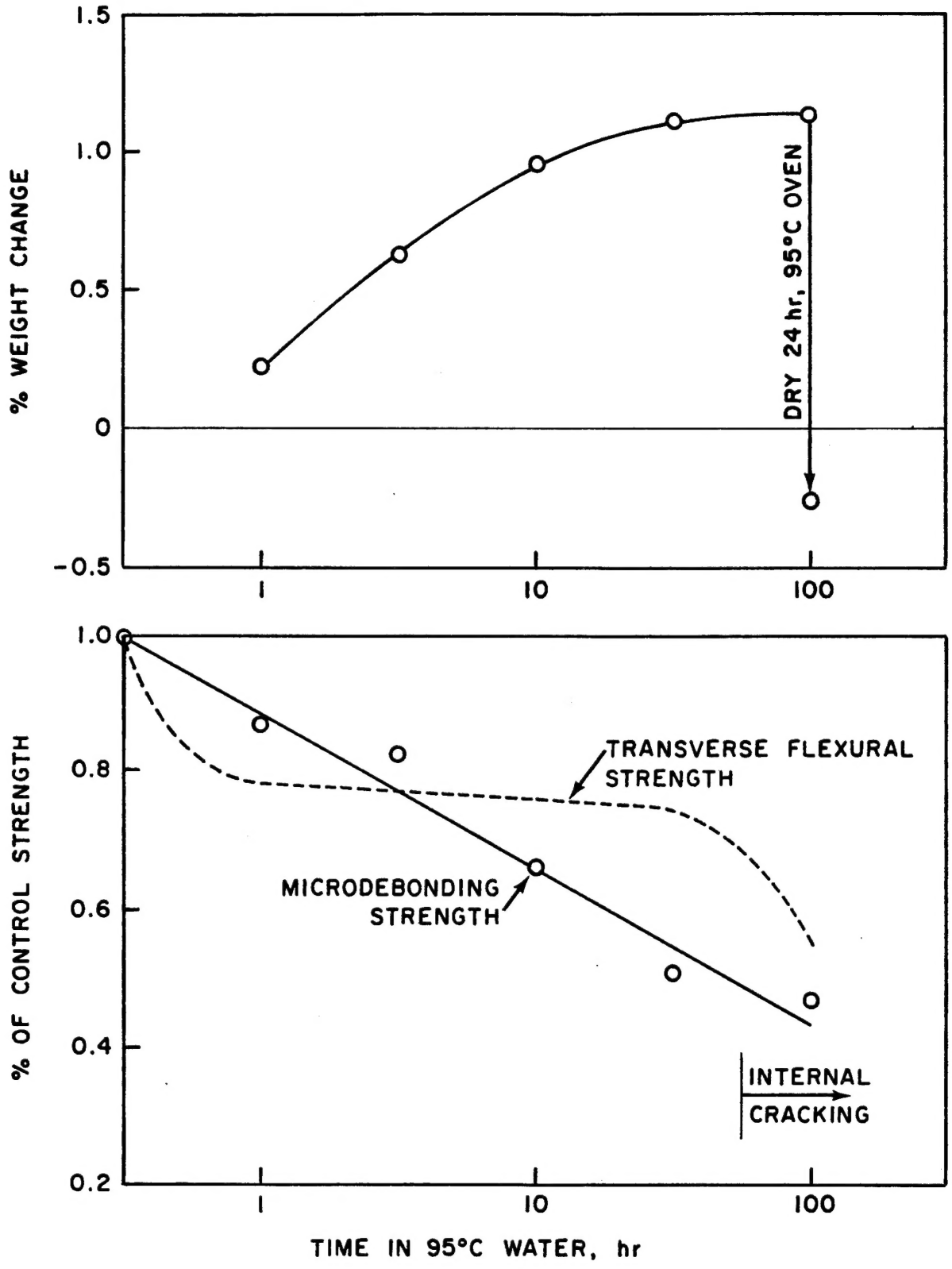


FIGURE 5.

EFFECT OF 95°C WATER CONDITIONING ON THE MICRODEBONDING STRENGTH AND TRANSVERSE FLEXURAL STRENGTH, GLASS/EPOXY, UNIDIRECTIONAL, DRIED BEFORE TESTING, MULTIFIBER DEBONDING, BATCH A.

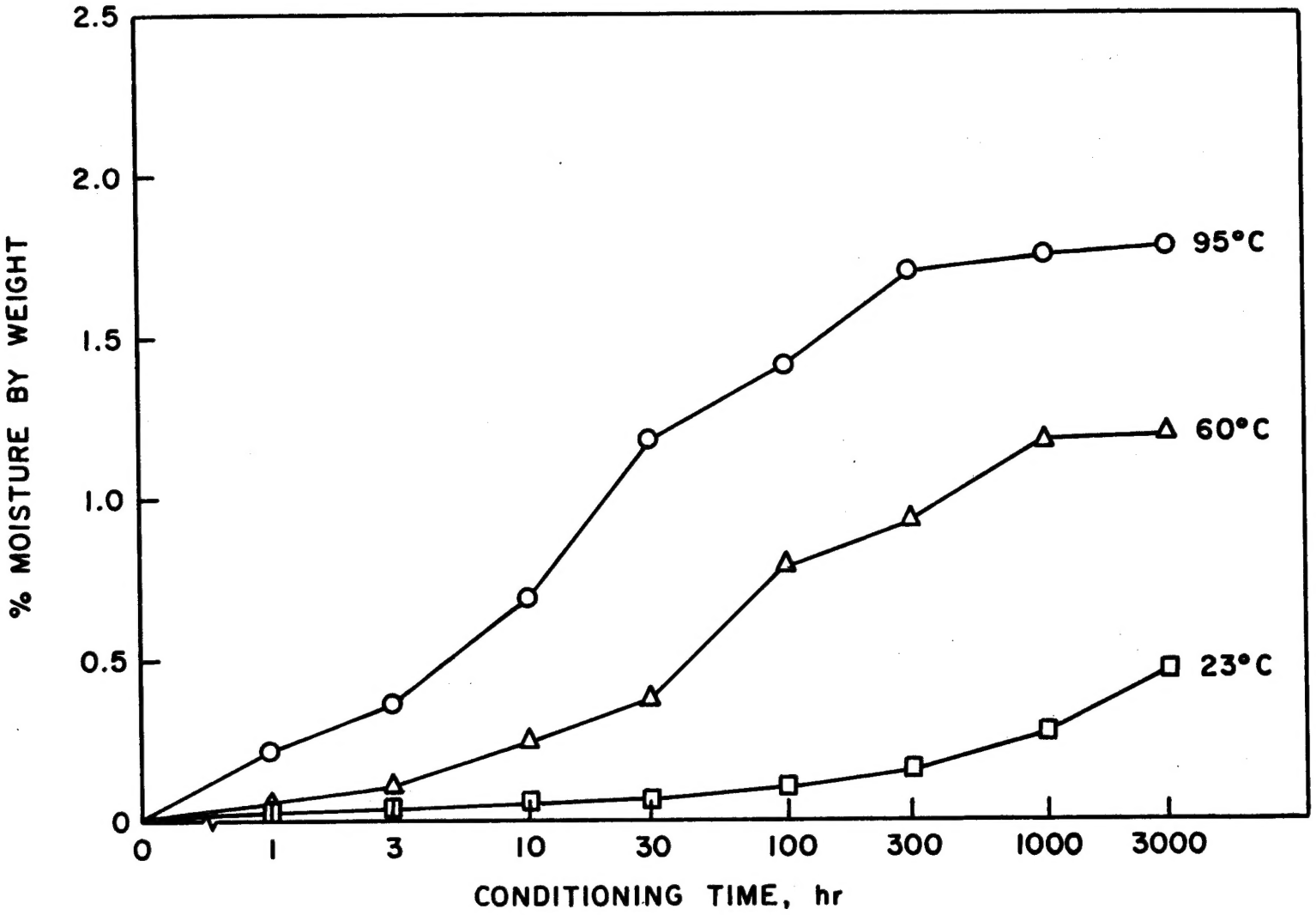


FIGURE 6.

MOISTURE GAIN vs. CONDITIONING TIME AT INDICATED TEMPERATURES, UNIDIRECTIONAL GLASS/EPOXY, BATCH B.

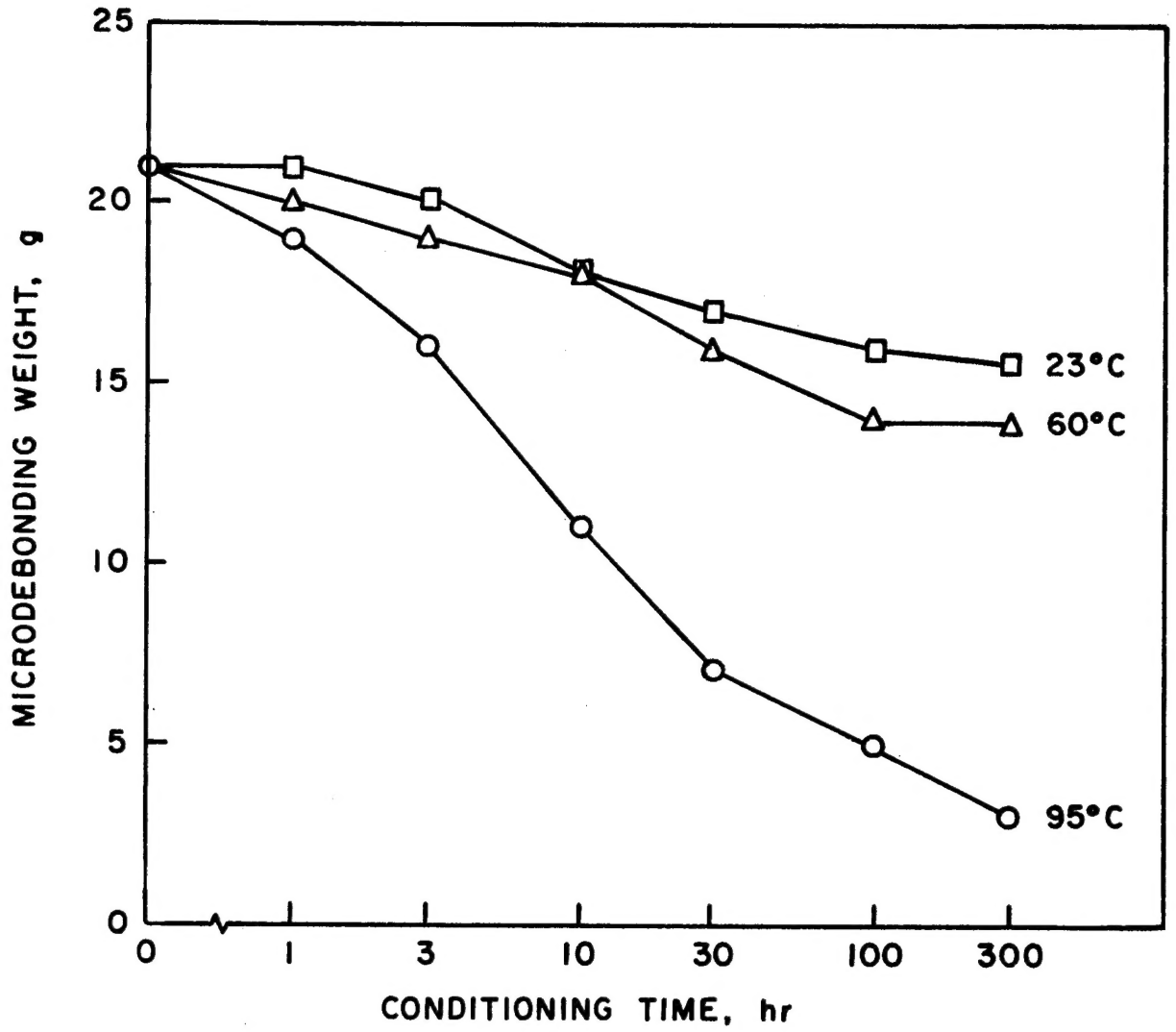


FIGURE 7.

MICRODEBONDING STRENGTH vs. CONDITIONING TIME IN DISTILLED WATER AT THE INDICATED TEMPERATURES, DRIED BEFORE TESTING, SINGLE FIBER DEBONDING, UNIDIRECTIONAL GLASS/EPOXY, BATCH B.

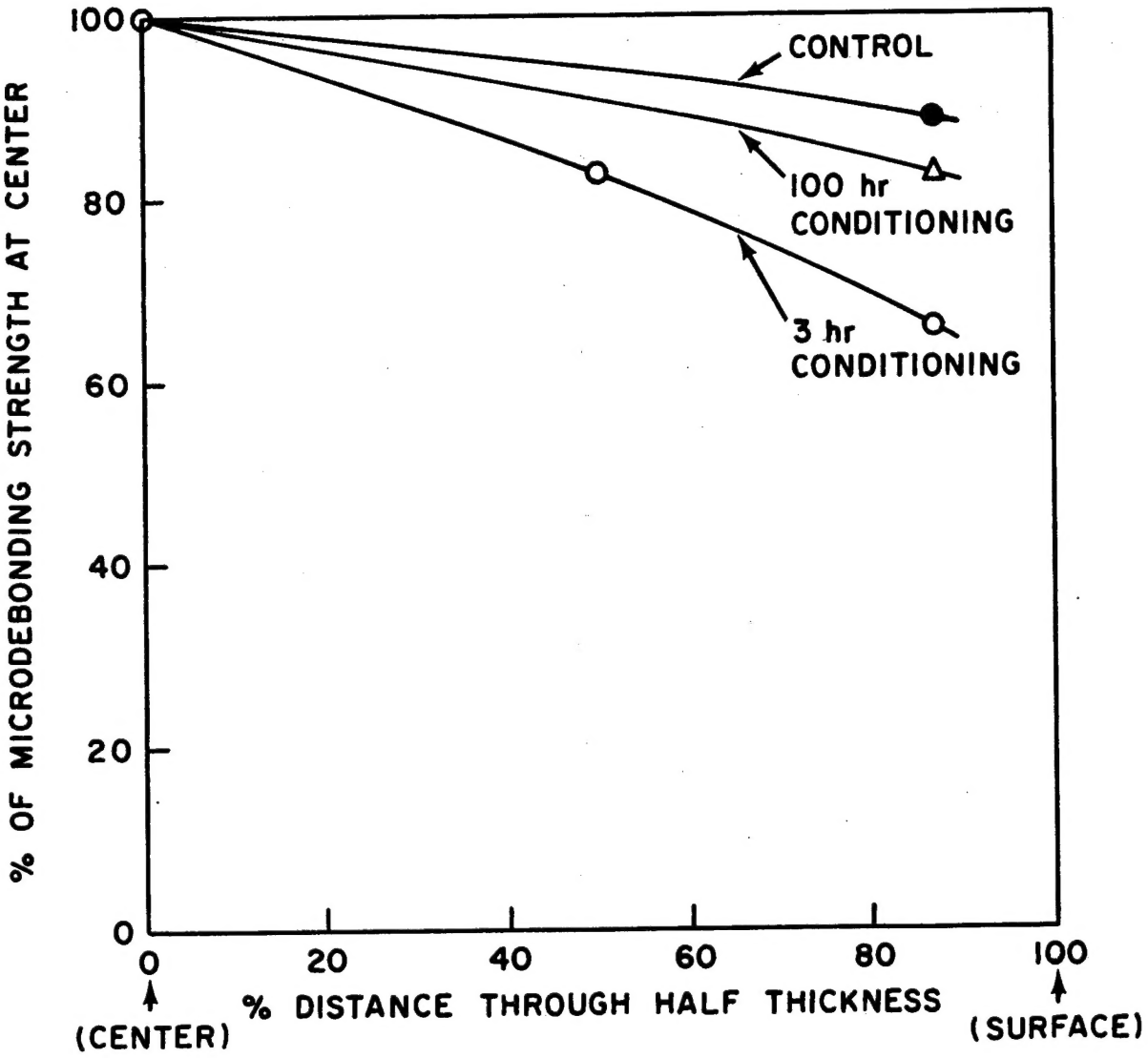


FIGURE 8.

MICRODEBONDING STRENGTH vs. DISTANCE THROUGH THICKNESS, BATCH A, CONDITIONED IN 95°C WATER.